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surface engineering

Novel advanced thermoset composites with intrinsic recyclability

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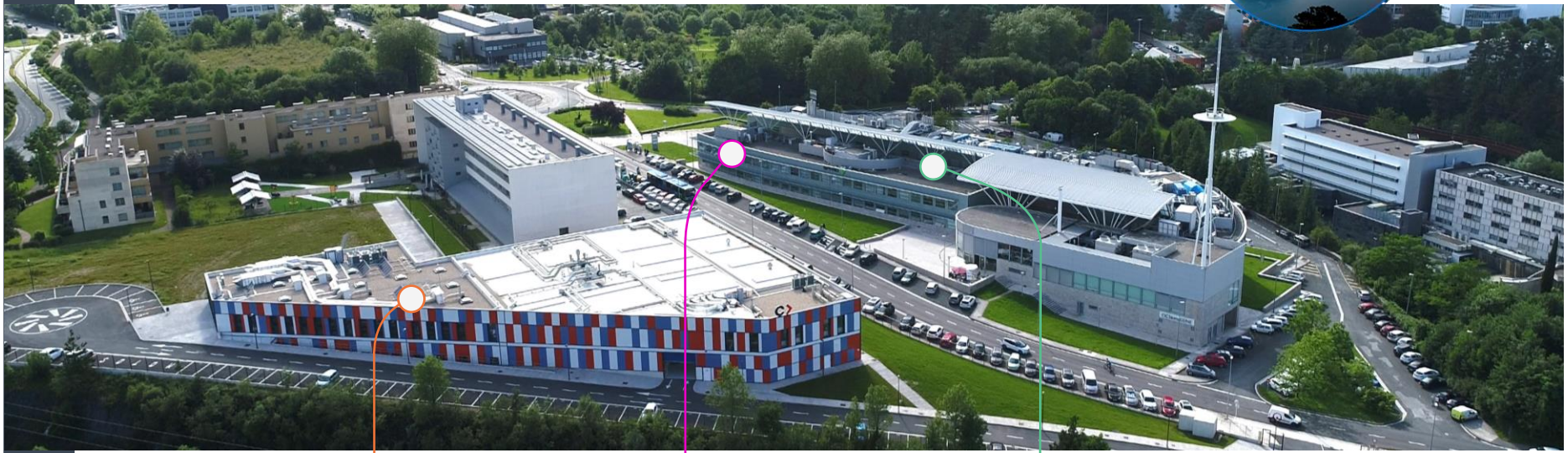
Innovations in Carbon Fibres and composite materials Recycling
Leitat, October 8th



surface engineering

nanomedicine

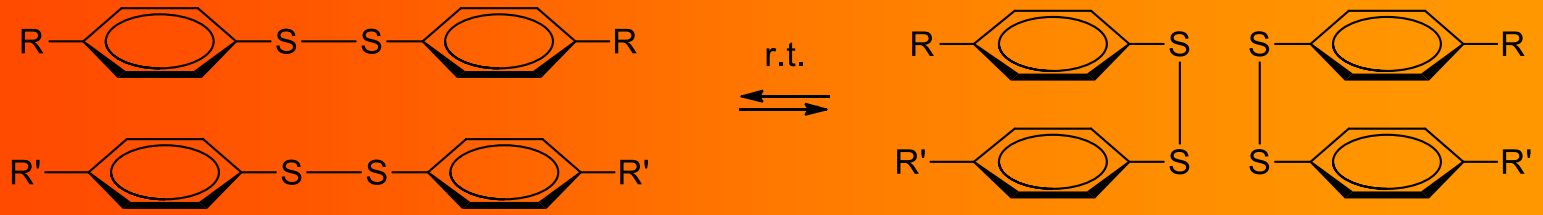
energy storage

Outline

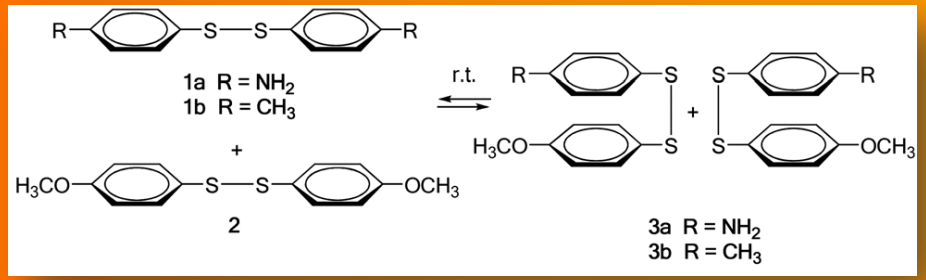
- ✓ Introduction & concepts
- ✓ Epoxy vitrimers based on aromatic disulfide
- ✓ Dynamic fibre reinforced thermoset composites
- ✓ AIRPOXY project
- ✓ Novel chemical recycling process of 3R composites

Introduction & Concepts

Vitrimers based on **aromatic disulfide exchange**

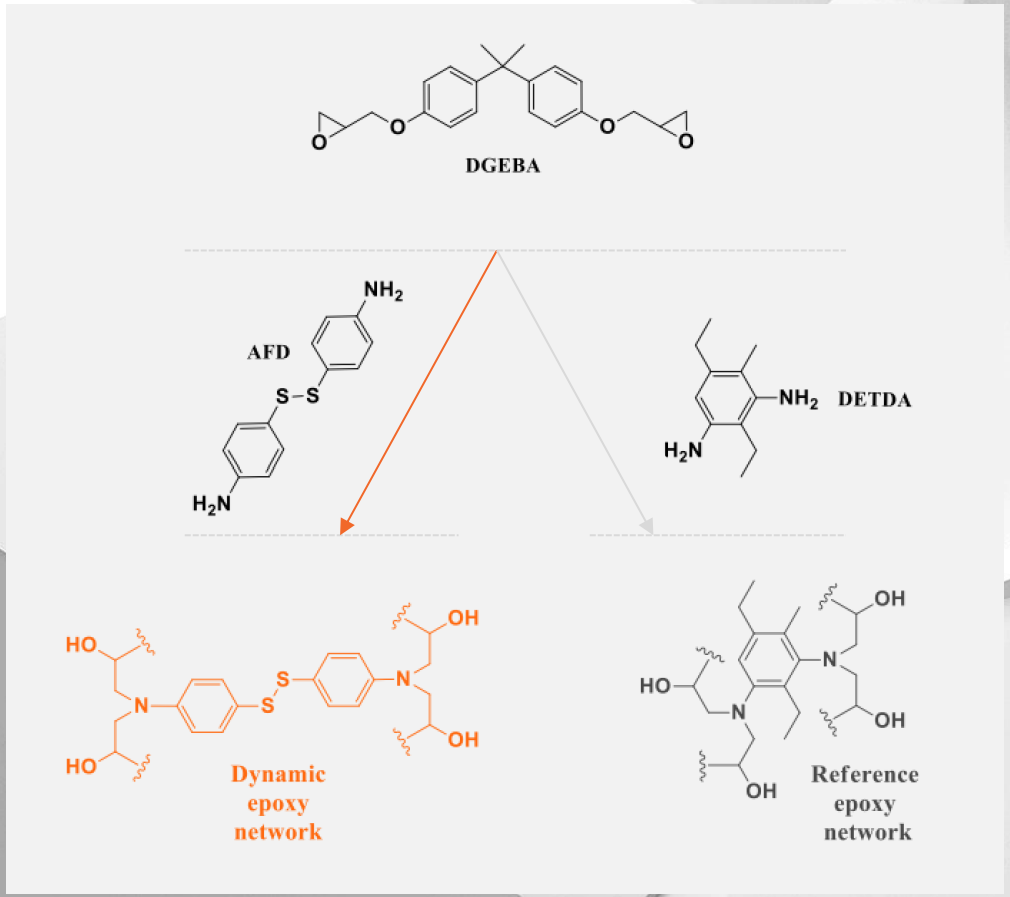


As a model metathesis reaction, we studied the equilibration of equimolar amounts of different aromatic disulfide mixtures by ^{1}H-NMR.



A. Rekondo, R. Martin, A. Ruiz de Luzuriaga, G. Cabañero, H. J. Grande and I. Odriozola, *Mater. Horiz.*, **2014**, *1*, 237–240

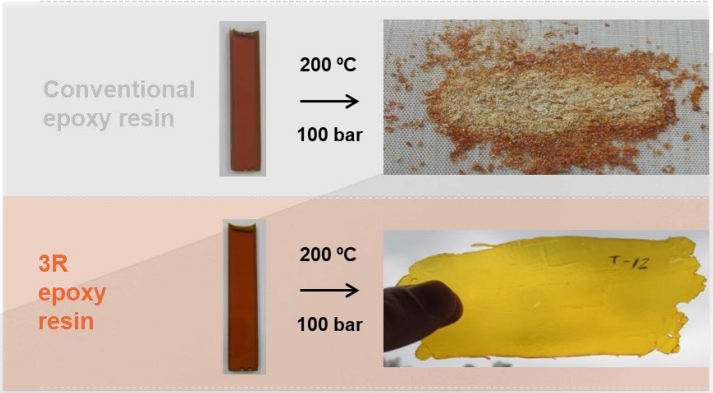
Epoxy vitrimers based on aromatic disulfide



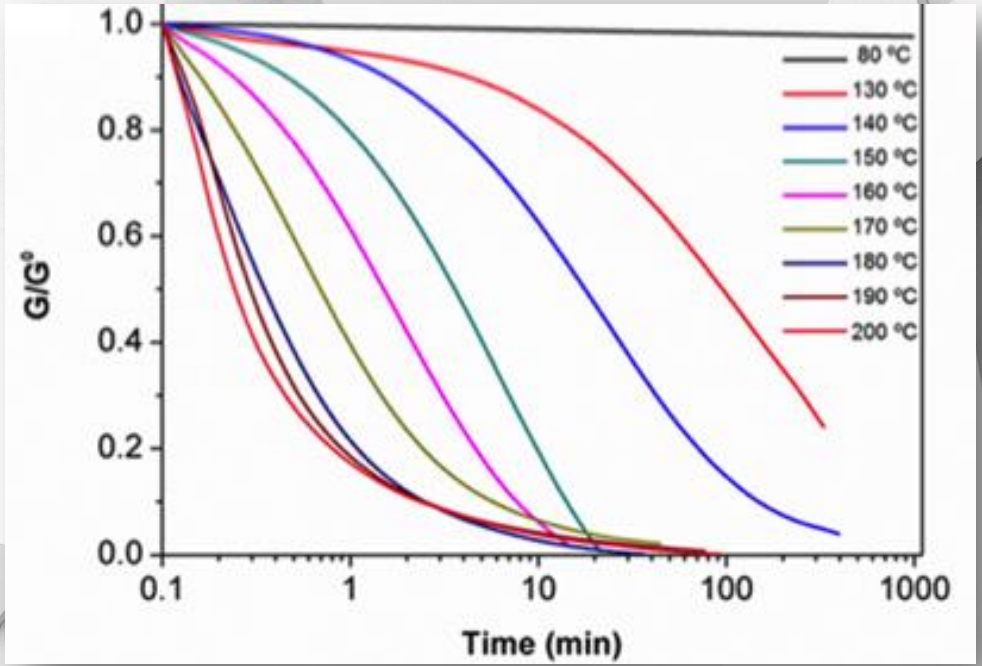
	Reference network	Dynamic network
Tg (DSC) [ºC]	127	**130**
Tg (DMA) [ºC]	130	**130**
Td [ºC]	350	**300**
E´(25ºC) [GPa]	2,5	**2,6**
E´(150ºC) [MPa]	20	**20**
Stress [MPa]	81	**88**
Strain [%]	7,3	**7,1**

Comparable thermal and mechanical properties using our dynamic hardener instead of a conventional hardener.

Epoxy vitrimers based on aromatic disulfide



Characterization of the stress relaxation by DMA



- At temperatures above Tg, **the dynamic epoxy network** is able to completely relax stress and flow.
- The obtained relaxation times ranged **from 3 hours at 130 ºC to 20 seconds at 200 ºC.**

A. Ruiz de Luzuriaga *et al.*, *Material Horizon*, **2016**, *3*, 241.

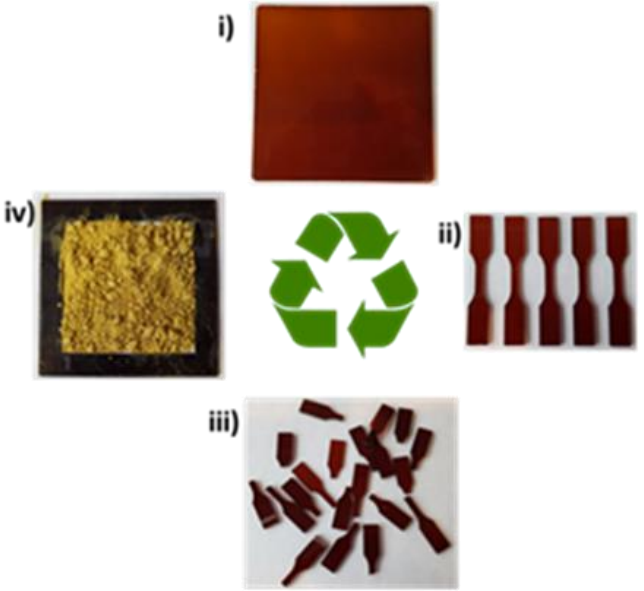
Epoxy vitrimers based on aromatic disulfide

The dynamic character of the epoxy network offers new challenges:

✓ Reparable thermoset resins

✓ a small scratch performed on a specimen was repaired just by applying heat a pressure with a household iron

✓ Recyclable thermoset resins



A. Ruiz de Luzuriaga *et al.*, *Material Horizon*, **2016**, *3*, 241.

Epoxy vitrimers based on aromatic disulfide

The dynamic character of the epoxy network offers new challenges:

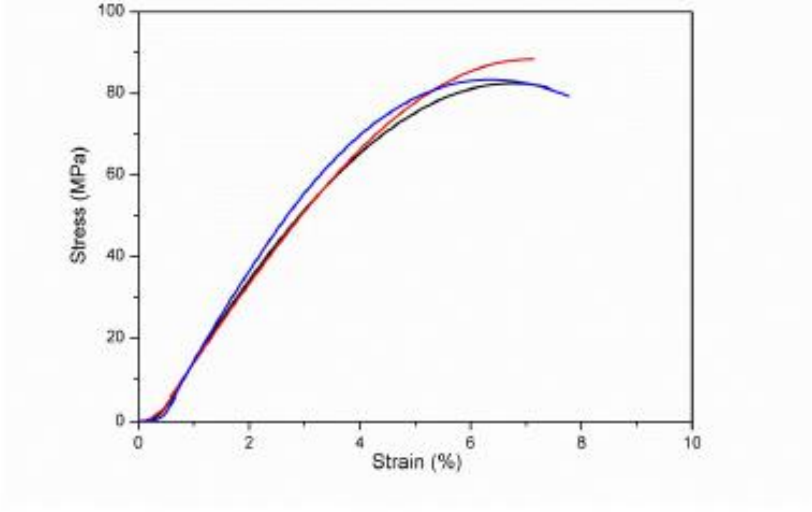
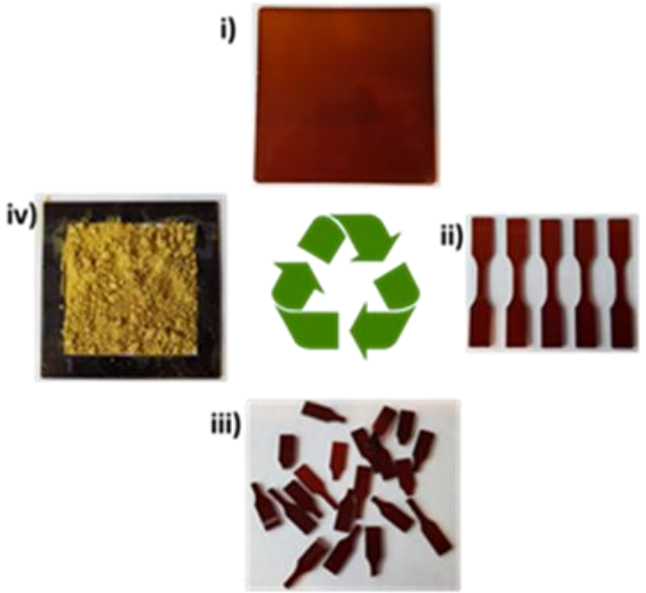


Figure S10. Stress vs. strain curves of reference epoxy network **4** (black trace), dynamic epoxy network **5** (red trace) and recycled dynamic epoxy network (blue trace).

✓ **Recyclable thermoset resins**



A. Ruiz de Luzuriaga *et al*., *Material Horizon*, **2016**, *3*, 241.

3R Composites

- A new generation of **Reprocessable, Repairable and Recyclable high-performance fibre-reinforced thermoset composites.**
- They can be manufactured following traditional methods but the resulting material can be reprocessed, repaired and recycled.

Dynamic fibre reinforced thermoset composites

> 3R Composites:

Reprocessing,

Repairing,

Recycling

3R composite laminates can be heated above the Tg and re-shaped in a few minutes applying pressure, which allows the thermoforming of cured 3R laminates to obtain 3D geometries, in a similar way to thermoplastic composites.

35%

reducing the manufacturing costs.

This technology enables the development of high production rate manufacturing processes for thermoset composites **reducing the manufacturing costs of thermoset CC parts by over 35% vs autoclave manufacturing.**

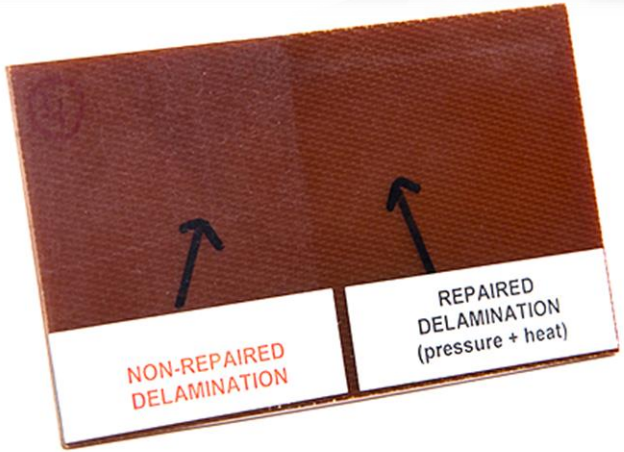
Dynamic fibre reinforced thermoset composites

> 3R Composites:

Reprocessing,

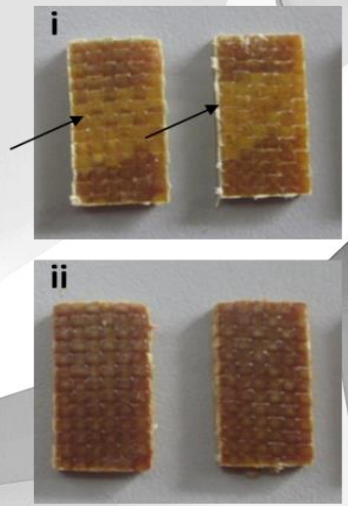
Repairing,

Recycling



Repair of damages based on resin/fibre delaminations and resin micro-cracks by applying heat and pressure to the damaged part.

ILSS: 37,2 ± 2,81 MPa



ILSS: 38,0 ± 2,4 MPa

50%

reducing the current costs of MRO.

This technology enables **to reduce the current costs of MRO associated to the reparation or replacement of thermoset CC parts by 50%** (nowadays damaged parts are often rejected due to the high costs and repair times of the traditional patch techniques).

Dynamic fibre reinforced thermoset composites

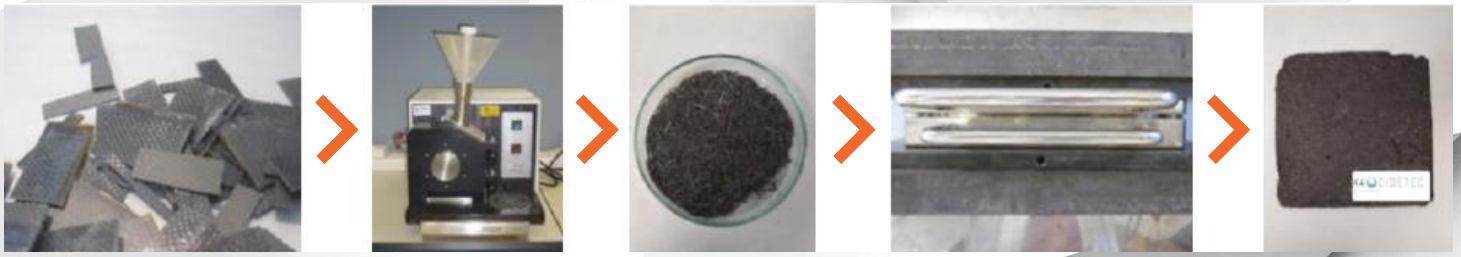
> 3R Composites:

Reprocessing,

Repairing,

Recycling

Mechanical recycling: the cured 3R composite can be ground into flakes or pellets which can then be reprocessed by heating 80ºC above the Tg and pressing, obtaining a new short fibre reinforced 3R composite.



This technology enables the valorisation of the scraps generated during the manufacturing of thermoset CC **offering 3 different environmental and industrial advantages**:

REDUCTION OF LANDFILL WASTE.

REDUCTION OF THE COSTS OF WASTE MANAGEMENT OF SCRAPS.

MANUFACTURING OF COST **COMPETITIVE AND SUSTAINABLE** NEW THERMOSET CC PARTS BASED ON VALORISED SCRAPS.

Dynamic fibre reinforced thermoset composites

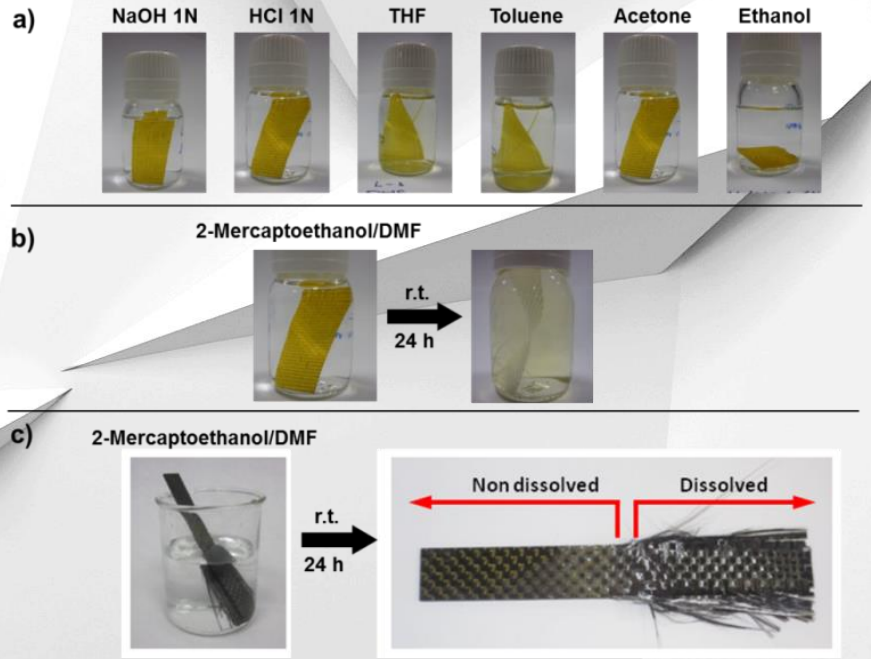
> 3R Composites:

Reprocessing,

Repairing,

Recycling

Chemical recycling: the 3R matrix can be completely disrupted by the addition of a specific chemical agent without affecting the reinforcement.

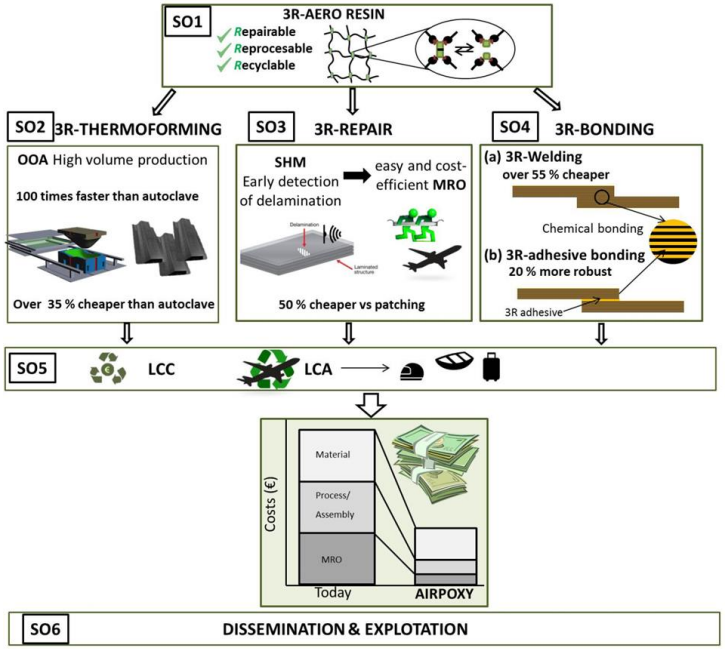


AIRPOXY

ThermoformAble, repaIrable and bondable smaRt ePOXY based composites for aero structures

HORIZON 2020

The aim of AIRPOXY is to **reduce the production and maintenance costs of composite parts in the aeronautic sector** by introducing a novel family of thermoset composites that preserve all the advantages of conventional thermosets, while showing new unprecedented features such as Re-processability, Reparability and Recyclability (3R – Composites).



ThermoformAble, repaIrable and bondable smaRt ePOXY based composites for aero structures

AIRPOXY

Our role.

CIDETEC acts as coordinator of the AIRPOXY project. Apart from this role, CIDETEC is in charge of several key technical tasks and activities, including:

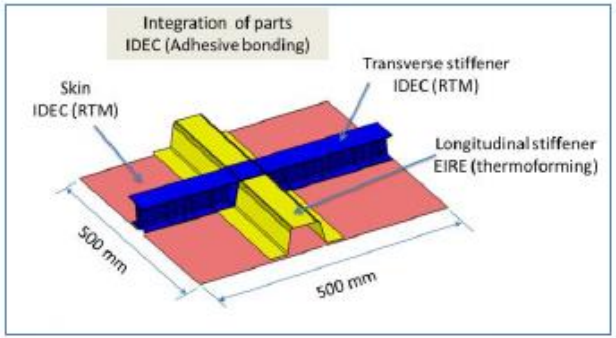
- Formulation of 3R resins for aerospace composites.

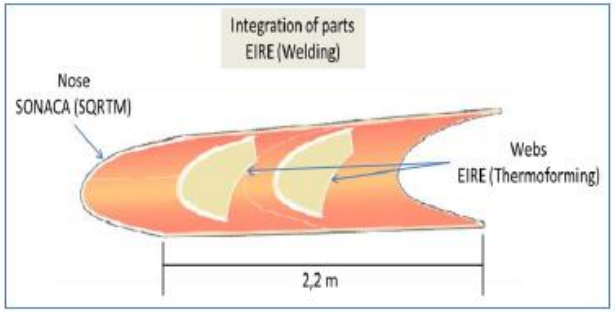
- Formulation of 3R adhesives for aerospace applications.

- Fabrication and characterization of 3R aerospace composites.

- Development of the 3R adhesive bonding process.

- Support the industrial partners regarding 3R technology.





Novel chemical recycling process of 3R composites

> 3R Composites:

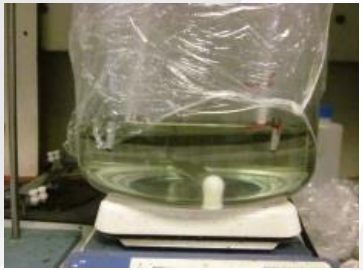
Reprocessing,

Repairing,

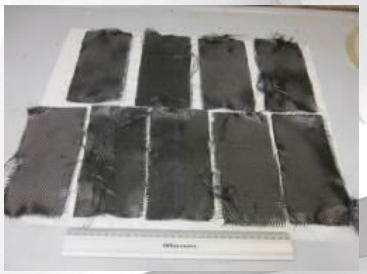
Recycling

Chemical recycling process

3R composite



Composite recycling



Recovered fabric

3R resin solution

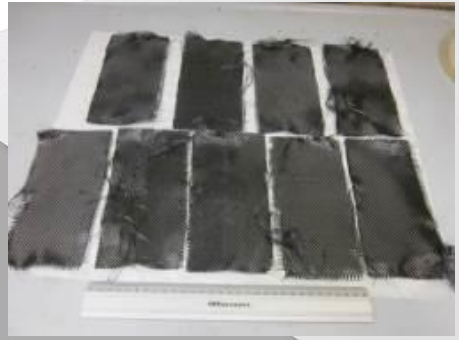
Novel chemical recycling process of 3R composites

> 3R Composites:

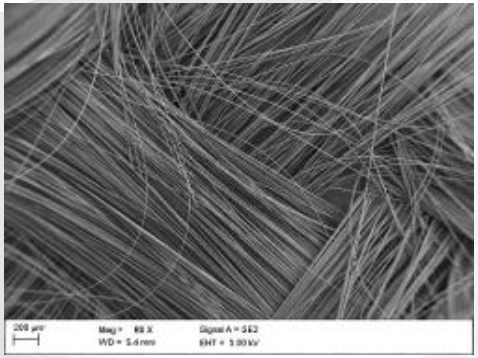
Reprocessing,

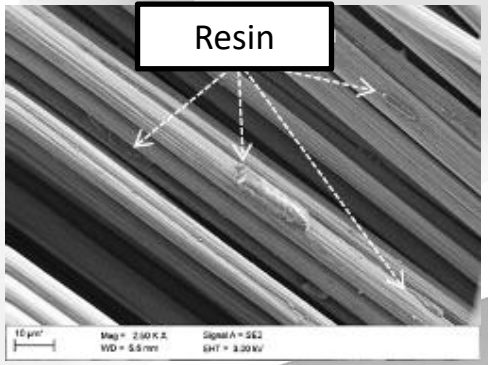
Repairing,

Recycling

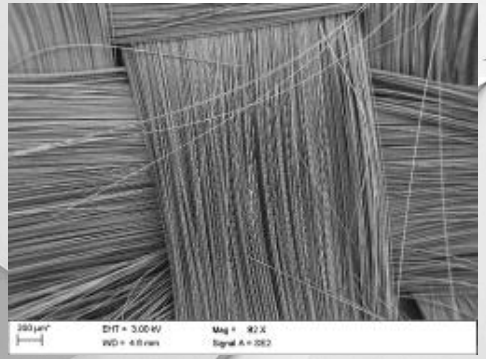


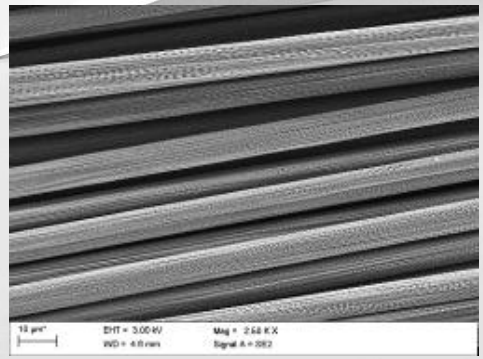
FE-SEM analysis of the recovered fabric





Recovered fabric after 4h





Recovered fabric after 8h

Novel chemical recycling process of 3R composites

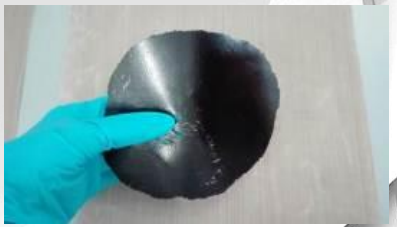
> 3R Composites:

Reprocessing,

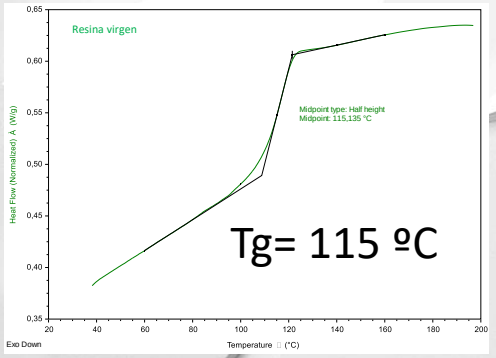
Repairing,

Recycling

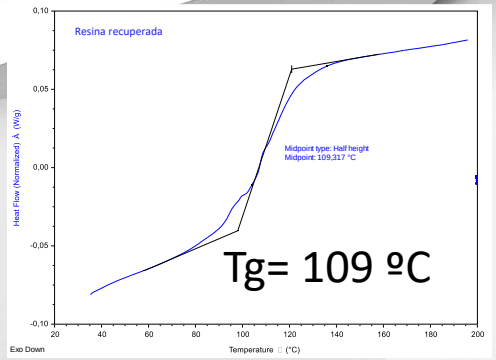
Recovered 3R resin



Virgin 3R resin



Recovered 3R resin



Thank you for your attention!

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